Development and Testing of a Miniaturized Explosives Preconcentrator for Use in a Man-portable Field Detection System

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Introduction

Sandia National Laboratories has utilized Department of Energy/Nuclear Emergency Search Team (DOE/NEST) funding to develop a miniaturized explosives preconcentrator to be used in a man-portable explosives detection system for field use. Current efforts are being directed toward packaging requirements that will allow true portability and a robust system. In addition, further testing is to be conducted using several common explosives in different field tests to determine overall system operability. This report will describe the development and testing to date of the miniaturized preconcentrator along with comparisons to the existing explosives preconcentrator used in the Sandia explosives detection portal developed for the Federal Aviation Administration (FAA) and DOE.

Preconcentrator Description and Requirements

As the name implies, the explosives preconcentrator's purpose is to concentrate explosive molecules for improved efficiency in detection. In general, a preconcentrator adsorbs explosive molecules from an inlet supply of air flowing at a high rate and relatively large volume, allowing those molecules of substances not of interest to pass through to an exhaust line. Then the adsorbing medium is heated quickly to desorb the explosive material into a low airflow, which is directed into a detector such as an ion mobility spectrometer (IMS) for trace detection. This process allows the explosive material present in the large air volume initially pulled into the preconcentrator to be concentrated into a much smaller air volume before it is delivered to the detector, greatly increasing the probability of detection when using a concentration sensitive detector such as an IMS. Figure 1 shows the general operation of a preconcentrator.

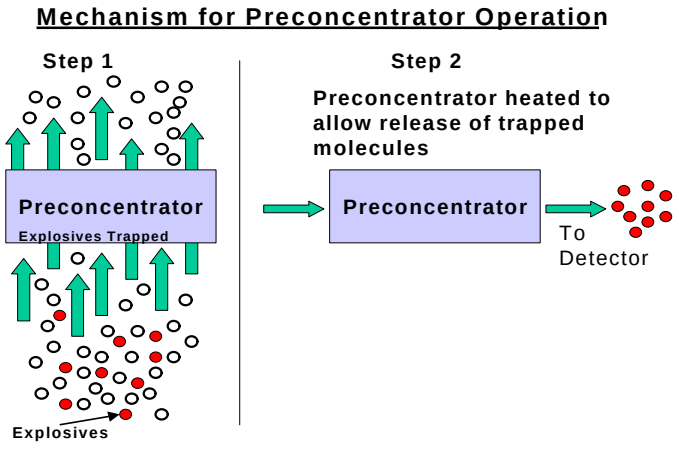

Figure 1. Preconcentrator Operation

In order to be utilized in a portable, field deployable detection system, the existing preconcentrator design in the Sandia personnel explosives detection portal required the following design modifications:

- Size miniaturization
- Portable, low DC power consumption operation
- Modular, field replaceable design
- High flow, high volume inlet for efficient vapor collection using miniaturized components

Description of Miniaturized Preconcentrator

The current prototype uses a metal felt mounted in a cylindrical housing as the adsorbing medium. A DC motor/impeller unit mounted directly beneath the preconcentrator adsorbing material generates airflow for sample collection. This flow pulls air from the suspect object or surface, through the 6 inch long, 1-inch diameter inlet tube, and then down through the metal felt screen where the explosive molecules adhere during the adsorption phase. The high inlet airflow is then stopped, and the metal screen is resistively heated to quickly desorb the explosive material into the gas phase. Subsequently, the desorbed molecules are entrained in the detector inlet airflow which in initiated at the beginning of the desorption cycle. The detector inlet airflow is typically 4 L/min, while the initial sampling air flow through the preconcentrator is as high as 185 L/min.

A significant difference between Sandia's portal preconcentrator and the miniaturized version is that no cross-flow is required in the latter during the desorption phase. This is due to the reduced volume inside the miniaturized preconcentrator and the close proximity of the detector inlet to the metal felt that serves as the adsorbing surface. Both pleated and unpleated metal felt screens were tested to determine effects on airflow, heating rates, and limits of detection (test results shown later).

A rechargeable, sealed lead acid battery rated at 12 volts and 7.2-ampere hours supplies power for the miniaturized preconcentrator. Typical current draw for the impeller motor and controller is 5 amperes during sampling. During the desorption cycle, the resistively heated metal screen can draw 20 amperes during operation. Given the small screen dimensions, this heating circuit requires energizing only 0.5 to 0.7 seconds per desorption cycle to maintain the proper desorb temperature. At the present time, the detector is powered by a separate battery pack attached to the detector case. Figure 2 shows the exposed pleats of the adsorbing material mounted in the miniaturized preconcentrator housing. A photograph of the handheld detector interfaced to the miniaturized preconcentrator complete with the inlet impeller is shown in Figure 3.

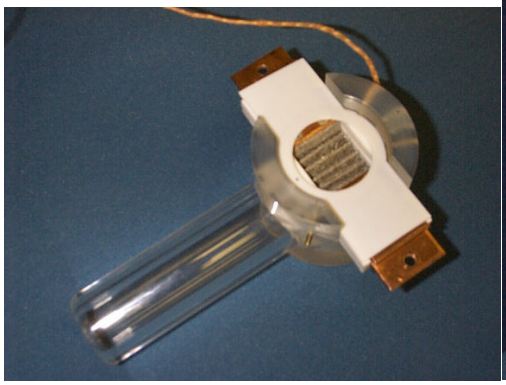

Figure 2. Inside the miniaturized preconcentrator, pleated adsorbing material exposed.

Figure 3. Preconcentrator interfaced to impeller and detector.

Preconcentrator Testing

Data will be presented from several tests conducted with the miniaturized preconcentrator. The explosives used were 2,4,6-trinitrotoluene (TNT) and cyclotrimethylenetrinitramine (RDX) desorbed from a 1-inch length of NiCr wire (hot-wire desorber). In this procedure, a known mass of explosive from a standard solution is deposited onto the bare wire, the wire is resistively heated, and the resulting explosive vapor is drawn into the preconcentrator inlet. Tests with the explosive pentaerythritol tetranitrate (PETN) have been limited thus far due to the potential molecular decomposition when desorbing PETN from a hot wire desorber. Future experiments will involve using a vapor generator as a PETN vapor source.

Volume Minimization

The original preconcentrator housing had an internal volume of 1.77 cubic inches. An adjustable piston was added to the inside of the housing in order to observe the effects of changing the internal volume of the preconcentrator for the adsorb/desorb cycles. With the piston in the lowest position, the internal adsorption/desorption volume was reduced to 0.51 cubic inches. The detector output was recorded after operating the preconcentrator with the piston in both the minimum and maximum volume positions. A TNT sample of one nanogram (ng) was used in these experiments, with the TNT being deposited onto the hot-wire in the form of one microliter of a 1 ng/microliter solution. Results are shown in Figure 4. Note that the reduction of preconcentrator volume is roughly proportional to the increase in detector output. These results suggest that the smaller preconcentrator volume allows the explosive material to be more highly concentrated when it enters the detector, thus leading to a substantial increase in the observed signal.

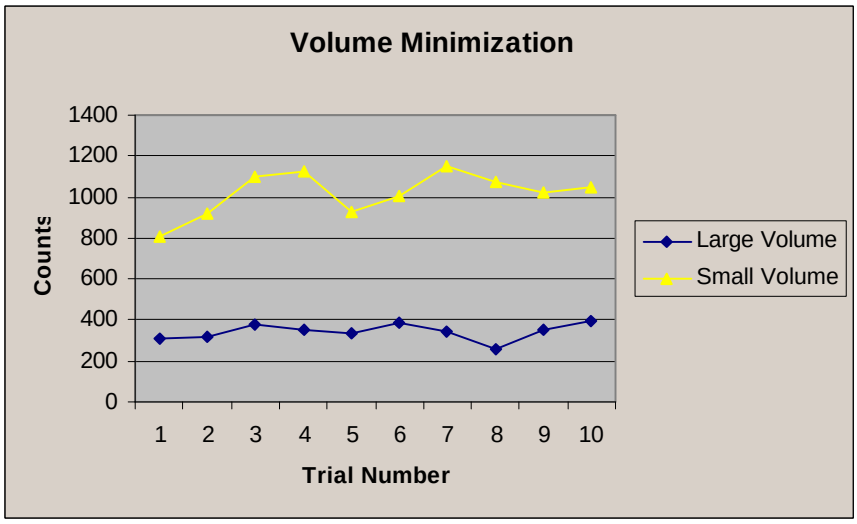

Figure 4. Chart shows preconcentrator volume effect on detector output.

Screen Configuration Tests

Several experiments were conducted to determine the optimum metal felt configuration, i.e., pleated or unpleated. Theoretically, the pleated material should trap more explosive molecules during an adsorb cycle because of the increased surface area. Furthermore, the pleated felt will allow a greater inlet flow for sampling than the unpleated felt of the same porosity since inlet flow is through the surface of the felt. A larger inlet flow is desirable for sample collection, but some data indicate that beyond a certain point inlet flow may pull the explosive molecules either completely through the adsorbing material or so deep into it that a single desorption cycle is insufficient for release to the detector. Figure 5 compares the detector output from 1 nanogram of TNT deposited directly onto the physical center of a pleated and an unpleated or flat metal felt. In these experiments any inconsistency in sample adsorption should be negated since the TNT solution was applied directly without inlet flow. The results of a similar comparison, but using the NiCr hot-wire for sample desorption into the inlet tube, are shown in Figure 6. Note that the detector signal is much greater for the unpleated felt in Figure 5, while the average signal is much more nearly the same for both felts in Figure 6. However, the scatter in the data is much smaller for the pleated felt in both figures. The data on the relative signal intensities from the two types of felts can be rationalized as follows. Desorption is more efficient from an unpleated felt, because the surface is flat and explosive molecules, once desorbed, can be pulled directly into the detector without undergoing further collisions with the felt surface. Thus in Figure 5, where only desorption is investigated, the unpleated felt gives consistently larger signals. However, when adsorbing material onto the felt, the pleated felt yields more efficient collection due to its larger surface area. Figure 6 shows that this advantage almost entirely offsets the disadvantage associated with desorption from the pleated felt, and thus the average signal intensities for the two

felts are nearly the same in this figure. Overall, the pleated felt seems preferable for consistent operation, due to the more consistent detector signal intensities that it produces.

Another series of tests was conducted to determine the effect of sample deposition on various sections of the adsorbing material. The metal felt was divided into nine sections, and a given mass of explosive was deposited with a syringe onto a different section for each adsorb/desorb cycle. Results showed that depositions furthest away from the detector inlet yielded output values similar to those directly in front of the inlet. Therefore, it can be concluded that the heating of the metal felt during the desorption cycle is fairly uniform, and that the detector inlet flow is sufficient to collect desorbed molecules within the entire preconcentrator volume.

Inlet Air Flow Velocity

The inlet air flow during the sampling or adsorbing cycle is supplied by an AMETEK brushless DC motor coupled to an impeller which is mounted on the back side of the metal felt. Tests were conducted to determine the effects on the detector output of varying the inlet flow velocity. The table below shows motor speed controller settings with corresponding inlet nozzle air velocities measured with an Omega digital anemometer (Model HH – 30).

Controller Setting (volts)	Input Airflow Velocity (L/sec.)
1	0.37
2	0.95
3	1.45
4	1.70
5	1.88

Table 1. Input Nozzle Air Velocities

The hot-wire desorber was used to vaporize 3 ng of RDX into the preconcentrator inlet airflow. Experiments were conducted at the five voltage settings with the resulting inlet flow velocities listed in the table above. When comparing the averaged results for the various inlet speeds, the slowest velocity yielded the largest detector output, while the fastest input flow showed a decrease in the average output by nearly a factor of 4. Results are shown in Figure 7. These results suggest the possible loss of sample through or around the metal felt at higher collection flow rates as mentioned previously.

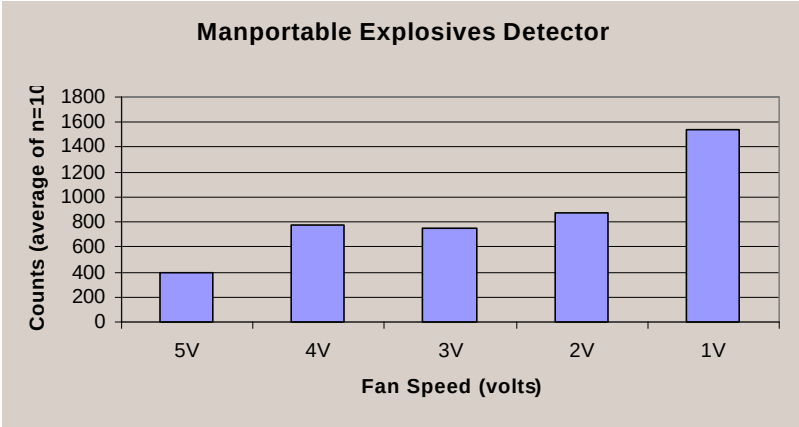

Figure 7. Detector output vs. inlet fan speed

Particulate Detection Tests

A series of experiments were conducted to determine the effect on detector output by operating the preconcentrator with metal felts rated at different porosity percentages. Three different metal felts were used, ranging in porosity from 77 % to 90%. The tests were performed by placing muslin patches doped with C-4 (prepared by the FAA) in front of the detection system inlet and comparing the resulting RDX alarms. Figure 8 shows the results using felt types 1, 2, and 3 with porosities of 90%, 87%, and 77% respectively.

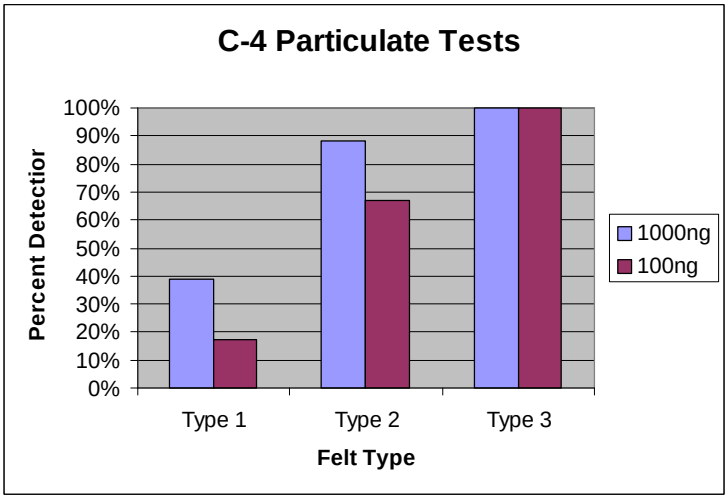

Figure 8. Detection percentage using different preconcentrator felts

Preconcentrator Efficiency

The miniature preconcentrator was tested in its present configuration to determine its preconcentration efficiency. The preconcentrator efficiency is the ratio of the number of counts from the detector for a given mass of explosive desorbed from a vapor inhalation to the number of counts for the same mass deposited directly onto the preconcentrator felt. For the first part of the tests, 1 ng of TNT was deposited by syringe directly onto the center of the preconcentrator metal felt. The felt was heated to 185° C and the resulting detector outlet was recorded. Then, using the same mass of explosive, TNT vapor was produced using a hot-wire desorber. The vapor was injected into the preconcentrator with an adsorption flow rate of 730 ft/min (corresponding to a 5 V controller setting), desorbed at 185° C, and the resulting detector output was recorded. The data for the two experiments are plotted in Figure 8. Calculating the average output over the ten trials for the two experiments gave a value for preconcentrator efficiency of 72% for 1 nanogram of TNT. Since part of the 28% loss may be due to the decomposition of TNT on the hot-wire during the initial desorption process, this figure represents a minimum value for the efficiency of the miniature preconcentrator.

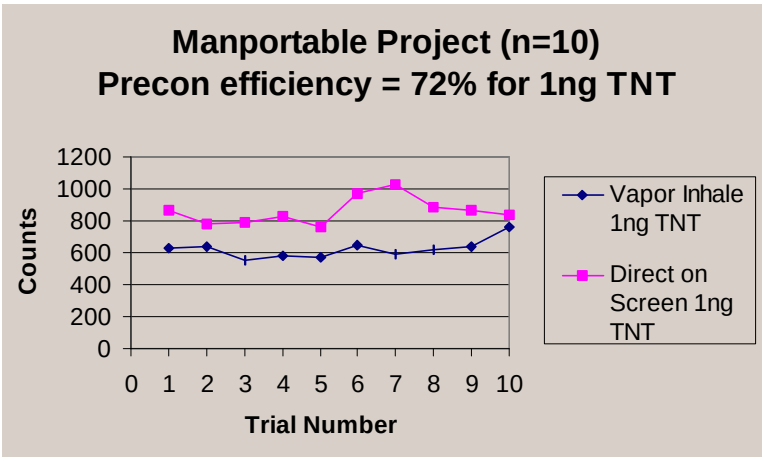

Figure 9. Preconcentrator efficiency for 1 nanogram of TNT.

Prototype Systems

Two man-portable explosives detection systems have been developed and are in the prototype stage. Both systems are shown interfaced to an Ion Track Instruments VaporTracer explosives/drug detector. The unit on the left is equipped to sample vapor using the preconcentrator described in this report. The system on the right is designed specifically for field analysis work, capable of swipe sampling using a fine textured metal felt and vapor sampling when the front-end attachment is removed.

Man-portable Explosive/Drug Detection Systems

Summary

As this report indicates, much work has been done to develop and test a prototype miniaturized preconcentrator for use with a man-portable, field deployable, explosives vapor detector. The basic configuration, including air flow and electronic components have been determined for the first prototype. Future efforts will involve work in the following areas:

1. Mapping the inlet and desorb flows within the preconcentrator housing and through the metal felt in order to minimize sample loss.

2. Experiment with changing the orientation of the pleats on the metal felt adsorbing material for greater adsorption efficiency. Also, efficiency at different flow rates will be evaluated with varying felt porosity.

3. Further miniaturization of components and interfacing with other handheld detectors.

4. Explore other applications for a chemical preconcentrator.

5. Improved packaging of the preconcentrator/detector system in order to produce a more robust, field-deployable system.

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Session V

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